



## **Analytical Laboratory**

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

#### **Order Summary Report**

Order Number:	J12020079			
Customer Name(s):	Bill Kennedy, Melonie Martin, Wayne	Chapman,	Tom Johnson	
Customer Address:	3195 Pine Hall Rd Mailcode: Belews Steam Station Belews Creek, NC 28012			
Lab Contact:	Jason C Perkins	Phone:	980-875-5348	
Report Authorized By: (Signature)		Date	e:	2/22/2012

#### **Program Comments:**

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

#### **Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted.

#### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

#### Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

## **Sample ID's & Descriptions:**

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2012002683	BELEWS	08-Feb-12 8:10 AM	R. HENDRICKS	FGD Purge Eff
2012002684	BELEWS	08-Feb-12 8:10 AM	R. HENDRICKS	EQ TANK EFF.
2012002685	BELEWS	08-Feb-12 8:15 AM	R. HENDRICKS	BIOREACTOR 1 INF.
2012002686	BELEWS	08-Feb-12 8:20 AM	R. HENDRICKS	BIOREACTOR 2 INF.
2012002687	BELEWS	08-Feb-12 8:25 AM	R. HENDRICKS	BIOREACTOR 2 EFF.
2012002688	BELEWS	08-Feb-12 8:00 AM	R. HENDRICKS	FILTER BLANK
2012002689	BELEWS	08-Feb-12 8:00 AM	R. HENDRICKS	Trip Blank
2012002690	BELEWS	08-Feb-12 1:15 PM	David Morris (Prism)	BIOREACTOR 1 INF.
2012002699	BELEWS	08-Feb-12 1:15 PM	David Morris (Prism)	HG BLANK BIOREACTOR 1 INF.
2012002700	BELEWS	08-Feb-12 1:25 PM	David Morris (Prism)	BIOREACTOR 2 INF.
2012002701	BELEWS	08-Feb-12 1:25 PM	David Morris (Prism)	Hg Blk BioReactor 2 Inf
2012002702	BELEWS	08-Feb-12 1:20 PM	David Morris (Prism)	BIOREACTOR 2 EFF.
2012002703	BELEWS	08-Feb-12 1:20 PM	David Morris (Prism)	Hg Blk BioReactor 2 Eff

#### **Technical Validation Review**

#### **Checklist:**

Mary Ann Ogle

Reviewed By:

	COC and .pdf report are in agreement with sample and analyses (compliance programs and procedure		<b>✓</b> Yes	☐ No
	All Results are less than the laboratory reporting lim	nits.	Yes	<b>✓</b> No
	All laboratory QA/QC requirements are acceptable.		✓ Yes	☐ No
	The Vendor Laboratories have been qualified by the Analytical Laboratory	Э	Yes	
Report S	ections Included:			
<b>✓</b> Jo	b Summary Report	✓ Sub-contr	acted Laborate	ory Results
<b>✓</b> Sa	mple Identification	☐ Customer	Specific Data	Sheets, Reports, & Documentation
<b>✓</b> Te	chnical Validation of Data Package	Customer	Database Ent	ries
<b>✓</b> Ar	alytical Laboratory Certificate of Analysis	✓ Chain of 0	Custody	
☐ Ar	alytical Laboratory QC Report	<b>✓</b> Electronic	: Data Delivera	able (EDD) Sent Separately

Date:

2/22/2012

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#### Order # J12020079

Site: FGD Purge Eff Sample #: 2012002683

Collection Date: 08-Feb-12 8:10 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC								
Bromide	100	mg/L		5	50	EPA 300.0		
MERCURY (COLD VAPOR)	IN WATER							
Mercury (Hg)	307	ug/L		5	100	EPA 245.1	10-Feb-12 10:21	AGIBBS
TOTAL RECOVERABLE ME	TALS BY ICP							
Boron (B)	194	mg/L		0.5	10	EPA 200.7	21-Feb-12 13:25	DJSULL1
Manganese (Mn)	7.08	mg/L		0.05	10	EPA 200.7	21-Feb-12 13:25	DJSULL1
DISSOLVED METALS BY IC	P-MS							
Manganese (Mn)	5660	ug/L		50	50	EPA 200.8	15-Feb-12 11:24	MHH7131
Selenium (Se)	275	ug/L		50	50	EPA 200.8	15-Feb-12 11:24	MHH7131
TOTAL RECOVERABLE ME	TALS BY ICP-MS							
Arsenic (As)	273	ug/L		10	10	EPA 200.8	15-Feb-12 10:51	MHH7131
Chromium (Cr)	299	ug/L		10	10	EPA 200.8	15-Feb-12 10:51	MHH7131
Copper (Cu)	160	ug/L		10	10	EPA 200.8	15-Feb-12 10:51	MHH7131
Nickel (Ni)	213	ug/L		10	10	EPA 200.8	15-Feb-12 10:51	MHH7131
Selenium (Se)	6280	ug/L		50	50	EPA 200.8	15-Feb-12 10:51	MHH7131
Silver (Ag)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 10:51	MHH7131
Zinc (Zn)	300	ug/L		10	10	EPA 200.8	15-Feb-12 10:51	MHH7131
SELENIUM SPECIATION								
Vendor Parameter	Complete				1	V_AS&C		
TOTAL DISSOLVED SOLIDS	<u> </u>							
TDS	16000	mg/L		200	1	SM2540C	09-Feb-12 15:30	TJA7067

Site: EQ TANK EFF. Sample #: 2012002684

Collection Date: 08-Feb-12 8:10 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY (COLD VAPOR) IN WATE	<u>:R</u>							
Mercury (Hg)	173	ug/L		2.5	50	EPA 245.1	10-Feb-12 10:42	AGIBBS
TOTAL RECOVERABLE METALS BY	<u> ICP</u>							
Boron (B)	182	mg/L		0.5	10	EPA 200.7	21-Feb-12 13:29	DJSULL1
Manganese (Mn)	6.87	mg/L		0.05	10	EPA 200.7	21-Feb-12 13:29	DJSULL1

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#### Order # J12020079

Site: EQ TANK EFF. Sample #: 2012002684

Collection Date: 08-Feb-12 8:10 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
DISSOLVED METALS BY ICP-MS								
Manganese (Mn)	5200	ug/L		50	50	EPA 200.8	15-Feb-12 11:27	MHH7131
Selenium (Se)	173	ug/L		10	10	EPA 200.8	15-Feb-12 11:27	MHH7131
TOTAL RECOVERABLE METALS B	Y ICP-MS							
Arsenic (As)	157	ug/L		10	10	EPA 200.8	15-Feb-12 10:54	MHH7131
Chromium (Cr)	180	ug/L		10	10	EPA 200.8	15-Feb-12 10:54	MHH7131
Copper (Cu)	96.9	ug/L		10	10	EPA 200.8	15-Feb-12 10:54	MHH7131
Nickel (Ni)	156	ug/L		10	10	EPA 200.8	15-Feb-12 10:54	MHH7131
Selenium (Se)	3810	ug/L		10	10	EPA 200.8	15-Feb-12 10:54	MHH7131
Silver (Ag)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 10:54	MHH7131
Zinc (Zn)	188	ug/L		10	10	EPA 200.8	15-Feb-12 10:54	MHH7131

Site: BIOREACTOR 1 INF. Sample #: 2012002685

Collection Date: 08-Feb-12 8:15 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC								
Bromide	93	mg/L		5	50	EPA 300.0	16-Feb-12 06:53	JAHERMA
TOTAL RECOVERABLE METALS BY	( ICP							
Boron (B)	162	mg/L		0.5	10	EPA 200.7	21-Feb-12 13:33	DJSULL1
Manganese (Mn)	3.43	mg/L		0.05	10	EPA 200.7	21-Feb-12 13:33	DJSULL1
DISSOLVED METALS BY ICP-MS								
Manganese (Mn)	3410	ug/L		10	10	EPA 200.8	15-Feb-12 11:30	MHH7131
Selenium (Se)	118	ug/L		10	10	EPA 200.8	15-Feb-12 11:30	MHH7131
TOTAL RECOVERABLE METALS BY	(ICP-MS							
Arsenic (As)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 10:57	MHH7131
Chromium (Cr)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 10:57	MHH7131
Copper (Cu)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 10:57	MHH7131
Nickel (Ni)	33.1	ug/L		10	10	EPA 200.8	15-Feb-12 10:57	MHH7131
Selenium (Se)	119	ug/L		10	10	EPA 200.8	15-Feb-12 10:57	MHH7131
Silver (Ag)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 10:57	MHH7131
Zinc (Zn)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 10:57	MHH7131
SELENIUM SPECIATION								
Vendor Parameter	Complete				1	V_AS&C		

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#### Order # J12020079

Site: BIOREACTOR 2 INF.

Sample #:

2012002686

Collection Date: 08-Feb-12 8:20 AM

Matrix:

OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS B	Y ICP							
Boron (B)	162	mg/L		0.5	10	EPA 200.7	21-Feb-12 13:37	DJSULL1
Manganese (Mn)	3.61	mg/L		0.05	10	EPA 200.7	21-Feb-12 13:37	DJSULL1
TOTAL RECOVERABLE METALS B	Y ICP-MS							
Arsenic (As)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:00	MHH7131
Chromium (Cr)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:00	MHH7131
Copper (Cu)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:00	MHH7131
Nickel (Ni)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:00	MHH7131
Selenium (Se)	24.5	ug/L		10	10	EPA 200.8	15-Feb-12 11:00	MHH7131
Silver (Ag)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:00	MHH7131
Zinc (Zn)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:00	MHH7131

Site: BIOREACTOR 2 EFF.

Vendor Parameter

Complete

Sample #:

V\_AS&C

2012002687

Collection Date: 08-Feb-12 8:25 AM

Matrix:

**OTHER** 

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC								
Bromide	89	mg/L		5	50	EPA 300.0	16-Feb-12 07:09	JAHERMA
MERCURY (COLD VAPOR) IN	I WATER							
Mercury (Hg)	< 1.00	ug/L		1	20	EPA 245.1	10-Feb-12 10:45	AGIBBS
TOTAL RECOVERABLE MET	ALS BY ICP							
Boron (B)	150	mg/L		0.5	10	EPA 200.7	21-Feb-12 13:41	DJSULL1
Manganese (Mn)	3.56	mg/L		0.05	10	EPA 200.7	21-Feb-12 13:41	DJSULL1
DISSOLVED METALS BY ICP	<u>-MS</u>							
Manganese (Mn)	3270	ug/L		10	10	EPA 200.8	15-Feb-12 11:33	MHH7131
Selenium (Se)	< 5.00	ug/L		5	5	EPA 200.8	15-Feb-12 11:33	MHH7131
TOTAL RECOVERABLE MET	ALS BY ICP-MS							
Arsenic (As)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:03	MHH7131
Chromium (Cr)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:03	MHH7131
Copper (Cu)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:03	MHH7131
Nickel (Ni)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:03	MHH7131
Selenium (Se)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:03	MHH7131
Silver (Ag)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:03	MHH7131
Zinc (Zn)	< 10.0	ug/L		10	10	EPA 200.8	15-Feb-12 11:03	MHH7131
SELENIUM SPECIATION								

## Certificate of Laboratory Analysis This report shall not be reproduced, except in full.

#### Order # J12020079

Site: FILTER BLANK Collection Date: 08-Feb-12	2 8:00 AM					Sample #: Matrix:	<b>2012002688</b> OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
DISSOLVED METALS BY ICP-N	<u>vis</u>							
Manganese (Mn)	1.36	ug/L		1	1	EPA 200.8	15-Feb-12 11:21	MHH7131
Selenium (Se)	1.41	ug/L		1	1	EPA 200.8	15-Feb-12 11:21	MHH7131
Site: Trip Blank						Sample #:	2012002689	
Collection Date: 08-Feb-12	2 8:00 AM					Matrix:	OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE META	LS BY ICP							
Boron (B)	< 0.050	mg/L		0.05	1	EPA 200.7	21-Feb-12 13:21	DJSULL1
Manganese (Mn)	< 0.005	mg/L		0.005	1	EPA 200.7	21-Feb-12 13:21	DJSULL1
TOTAL RECOVERABLE META	LS BY ICP-MS							
Arsenic (As)	< 1.00	ug/L		1	1	EPA 200.8	15-Feb-12 10:48	MHH713
Chromium (Cr)	< 1.00	ug/L		1	1	EPA 200.8	15-Feb-12 10:48	MHH713
Copper (Cu)	2.29	ug/L		1	1	EPA 200.8	15-Feb-12 10:48	MHH713
Nickel (Ni)	< 1.00	ug/L		1	1	EPA 200.8	15-Feb-12 10:48	MHH713
Selenium (Se)	< 1.00	ug/L		1	1	EPA 200.8	15-Feb-12 10:48	MHH713
Silver (Ag)	1.37	ug/L		1	1	EPA 200.8	15-Feb-12 10:48	MHH713
Zinc (Zn)	1.76	ug/L		1	1	EPA 200.8	15-Feb-12 10:48	MHH7131
SELENIUM SPECIATION								
Vendor Parameter	Complete				1	V_AS&C		
Site: BIOREACTOR 1 IN	NF.					Sample #:	2012002690	
Collection Date: 08-Feb-12	2 1:15 PM					Matrix:	OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY 1631								
Vendor Parameter	Complete				1	V_BRAND		
Site: HG BLANK BIORE Collection Date: 08-Feb-12						Sample #: Matrix:	<b>2012002699</b> OTHER	
Anglista	Page-It	lu!t=	Qualifican	וחם	DE	لد - حالم 84	Anglusia Deta/Tim-	Amelia
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY 1631 Vendor Parameter	Complete				1	V_BRAND		

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#### Order # J12020079

Site: BIOREACTOR 2	INF.					Sample #:	2012002700	
Collection Date: 08-Feb-	12 1:25 PM					Matrix:	OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY 1631								
Vendor Parameter	Complete				1	V_BRAND		
Site: Hg Blk BioReacto	or 2 Inf					Sample #:	2012002701	
Collection Date: 08-Feb-	12 1:25 PM					Matrix:	OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY 1631								
Vendor Parameter	Complete				1	V_BRAND		
Site: BIOREACTOR 2	EFF.					Sample #:	2012002702	
Collection Date: 08-Feb-	12 1:20 PM					Matrix:	OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY 1631								
Vendor Parameter	Complete				1	V_BRAND		
Site: Hg Blk BioReacto	or 2 Eff					Sample #:	2012002703	
Collection Date: 08-Feb-	12 1:20 PM					Matrix:	OTHER	
Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY 1631								
Vendor Parameter	Complete				1	V_BRAND		



18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

February 21, 2012

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Belews – FGD WWTS (Bi-Monthly Wed-Sampling) (LIMS # J12020079)

Dear Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for selenium speciation analysis on February 9, 2012. The samples were received in a sealed cooler at -0.5°C on February 10, 2012. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma kinetic energy discrimination cell mass spectrometry (IC-ICP-KED-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

#### Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078

Project: Belews – FGD WWTS (Bi-Monthly Wed-Sampling) (LIMS # J12020079)

February 21, 2012

#### 1. Sample Reception

Four (4) aqueous samples in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on February 9, 2012. The samples were received on February 10, 2012 in a sealed container at -0.5°C.

The samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and was designated a discrete sample identifier. An aliquot of each sample was filtered (0.45 µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80 °C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma kinetic energy discrimination mass spectrometry (IC-ICP-KED-MS).

#### 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Selenium Speciation Analysis by IC-ICP-KED-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

#### 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are

standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimal interval of every ten analytical runs.

<u>Selenium Speciation Analysis by IC-ICP-KED-MS</u> Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma kinetic energy discrimination mass spectrometry (IC-ICP-KED-MS) on February 16, 2012. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (KED) containing hydrogen gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

#### 4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with this sample were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

## Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS (Bi-Monthly-Wed-Sampling) Contact: Jay Perkins LIMS #J12020079

Date: February 21, 2012 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

#### Sample Results

						Unknown Se
Sample ID	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Species (n)
FGD Purge Eff	124	64.0	ND (<0.86)	1.96	ND (<0.59)	0 (0)
BioReactor 1 Inf	26.0	60.6	ND (<0.21)	3.50	ND (<0.15)	0 (0)
BioReactor 2 Eff	0.42	ND (<0.12)	ND (<0.21)	ND (<0.15)	ND (<0.15)	0 (0)
Metals Trip Blk	ND (<0.022)	ND (<0.024)	ND (<0.043)	ND (<0.030)	ND (<0.030)	0 (0)

All results reflect the applied dilution and are reported in µg/L

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

n = number of unknown Se species observed

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS (Bi-Monthly-Wed-Sampling) Contact: Jay Perkins LIMS #J12020079

Date: February 21, 2012 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

#### **Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 10x	eMDL 50x	eMDL 200x
Se(IV)	0.010	0.017	0.010	0.011	0.012	0.003	0.002	0.022	0.11	0.43
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	0.024	0.12	0.48
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.004	0.043	0.21	0.86
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.003	0.030	0.15	0.59
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.003	0.030	0.15	0.59

eMDL = Estimated Method Detection Limit

#### **Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Se(IV)	LCS	9.57	9.48	99.1
Se(VI)	LCS	9.48	9.04	95.4
SeCN	LCS	8.92	8.44	94.6
MeSe(IV)	LCS	6.47	6.07	93.8
SeMe	LCS	9.32	8.70	93.3

<sup>\*</sup>Please see narrative regarding eMDL calculations

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS (Bi-Monthly-Wed-Sampling) Contact: Jay Perkins LIMS #J12020079

Date: February 21, 2012 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

#### **Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Se(IV)	Batch QC	0.69	0.62	0.7	10.3
Se(VI)	Batch QC	ND (<0.48)	ND (<0.48)	NC	NC
SeCN	Batch QC	4.61	4.36	4.5	5.4
MeSe(IV)	Batch QC	ND (<0.59)	ND (<0.59)	NC	NC
SeMe	Batch QC	ND (<0.59)	ND (<0.59)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

#### **Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate**

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Se(IV)	Batch QC	1112	1186	106.7	1112	1185	106.6	0.1
Se(VI)	Batch QC	1009	1063	105.4	1009	1056	104.7	0.6
SeCN	Batch QC	915.0	955.7	104.0	915.0	946.6	103.0	1.0

Comments	11)Seal/Locked By	eyseallLocked By	7)Relinquished By	5)Relinquished By	3) Relinguished By	1) Relinquished By	1	88		9.8	8	20	190000	<sup>11</sup> Lab ID	LAB USE ONLY	8)Oper. Unit:	5)Business ∪nit:			ge 16 o	127 10	
	Date/Time	J-9-/ Date/Time	2-9-/2	Date/Time	Which 2 F12 Date Fine	Mr Handle Datestime	SIBIALA arecono senso se estado como como como como como como como co	Filte	Bio Richard	BioRea	1303 OH Transmission of the Contract of the Co	EQTa	<del></del>	<sup>13</sup> Sample De	Se Speciation Bottle	9)Res. Type:	6)Process:	Bill Kennedy, Melonie Martin, Wayne Chapman, Tom Johnson **	Belews - FGD	Fax: (704) 875-4349	Duke Er Mail	CHAIN OF CUS
7	12)Seal/Lock Opened By	e bened	8)Accepted By  Agreem	6/Accepted By:	1/3> a) Magneti By	8-/2 27.Acceptadi.By	Wetals Trip Bik 2/8 0800	3/8	BioReactor-2-Tifficularian managed placements of the second secon	BioReactor 2 Inf 2/8 0820	HICH REDECTOR FIRM	8/8	FGB Perge Eff 4/8 08/0	-		10)Reso. Center: Custome	Mail Code: MR#	4)Fax No: PO#133241	0.	5245 Logged By 5255	ical Laboratory (Building 7405) Ferry Rd C 20078	CUSTODY RECORD AND ANALY
	3y Date/Time	By Date/Time	Meries -0 50 Shoffa		FMOR 2-8-12 Date/Ime	Man 2 \$12 Date Time	TRANS CONTRACTOR OF STATE OF S	00	Commence of the second	00		10	O H. Kendricks	Signature 17 Co	rab S	Customer to complete all	yses	2=H <sub>2</sub> SO <sub>4</sub> 3=HNG <sub>5</sub> 4=1Ce 5=Nome 4	Coopler Temp (C)	150 0 2 cm	Analytical Laboratory Use Only Samples Matrix: OTHER Originat From	ANALYSIS REQUEST FORM
	2-16	*Other * Add. Cost Will Apply	9 48 H	*7 Days	1530 14 Days	22Requested Turnaround	Para kuwa pala mana mana menangan menangan dalam 1		main and the state of the state					Me Mn	tals* , Se, s	ation - ver	e filled	3,4	RCRA Waste	SAMPLE PROGRAM Ground COPY to CLIENT Water NPDES ( Drinking Water )	NO L	FORM TUSK O



February 15, 2012

Duke Energy ATTN: Jay Perkins Scientific Support-Laboratory 13339 Hagers Ferry Road Huntersville NC 28078 jcperkins@duke-energy.com labcustomer@duke-energy.com

RE: Project DUK-HV1201 Client Project: J12020079

Dear Mr. Perkins,

On February 10, 2012, Brooks Rand Labs (BRL) received three (3) wastewater samples and three (3) corresponding field blanks. Samples were logged-in for total mercury (Hg) analysis. All samples were received, prepared, analyzed, and stored according to BRL SOPs and EPA methodology.

The results were blank-corrected as described in the calculations section of the applicable SOP(s) and may be evaluated using adjusted reporting limits to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific detection limits and other details.

No qualification of the data was warranted, aside from concentration qualifiers, and all associated quality control sample results met the acceptance criteria.

BRL, an accredited laboratory, certifies the reported results of all analyses for which BRL is NELAP accredited meet all NELAP requirements. For more details, see the *Report Information* page of the report. Please feel free to contact me if you have any questions regarding this report.

Sincerely,

Tiffany Stilwater Project Manager

tiffany@brooksrand.com

tilwate

Project ID: DUK-HV1201 PM: Tiffany Stilwater



Analytical Lab Page 18 of 27 Client PM: Jay Perkins

**Client PO: 141391** 

### Report Information

#### **Laboratory Accreditation**

BRL is accredited by the National Environmental Laboratory Accreditation Program (NELAP) through the State of Florida Department of Health, Bureau of Laboratories (E87982) and is certified to perform many environmental analyses. BRL is also certified by many other states to perform environmental analyses. For a current list of our accreditations /certifications, please visit our website at <a href="http://www.brooksrand.com/default.asp?contentID=586">http://www.brooksrand.com/default.asp?contentID=586</a>. Results reported relate only to the samples listed in the report.

#### **Field Quality Control Samples**

Please be notified that certain EPA methods require the collection of field quality control samples of an appropriate type and frequency; failure to do so is considered a deviation from some methods and for compliance purposes should only be done with the approval of regulatory authorities. Please see the specific EPA methods for details regarding required field quality control samples.

#### **Common Abbreviations**

BLK	method blank	MS	matrix spike
BRL	Brooks Rand Labs	MSD	matrix spike duplicate
BS	laboratory fortified blank	ND	non-detect
CAL	calibration standard	NR	non-reportable
CCV	continuing calibration verification	PS	post preparation spike
COC	chain of custody record	REC	percent recovery
CRM	certified reference material	RPD	relative percent difference
D	dissolved fraction	RSD	relative standard deviation
DUP	duplicate	SCV	secondary calibration verification
ICV	initial calibration verification	SOP	standard operating procedure
MDL	method detection limit	SRM	standard reference material
MRL	method reporting limit	Т	total recoverable fraction

#### **Definition of Data Qualifiers**

(Effective 9/23/09)

- Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate. В
- An estimated value due to the presence of interferences. A full explanation is presented in the narrative. Ε
- Holding time and/or preservation requirements not met. Result is estimated. Н
- Estimated value. A full explanation is presented in the narrative. J
- Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated. J-M
- J-N Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
- Duplicate precision (RPD) was not within acceptance criteria. Result is estimated. M
- Spike recovery was not within acceptance criteria. Result is estimated. Ν
- Rejected, unusable value. A full explanation is presented in the narrative. R
- U Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
- X Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

These qualifiers are based on those previously utilized by Brooks Rand, Ltd., those found in the EPA SOW ILM03.0, Exhibit B, Section III, pg. B-18, and the USEPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses; USEPA; July 2002. These supersede all previous qualifiers ever employed by BRL.

Project ID: DUK-HV1201 PM: Tiffany Stilwater



Analytical Lab Page 19 of 27 Client PM: Jay Perkins

**Client PO: 141391** 

## Sample Information

Sample	Lab ID	<b>Report Matrix</b>	Туре	Sampled	Received
BioReactor 1 Inf	1206023-01	Influent	Sample	02/08/2012	02/10/2012
Hg Blk BioReactor 1 Inf	1206023-02	DIW	Field Blank	02/08/2012	02/10/2012
BioReactor 2 Inf	1206023-03	Influent	QC Sample	02/08/2012	02/10/2012
Hg Blk BioReactor 2 Inf	1206023-04	DIW	Field Blank	02/08/2012	02/10/2012
BioReactor 2 Eff	1206023-05	Effluent	Sample	02/08/2012	02/10/2012
Hg Blk BioReactor 2 Eff	1206023-06	DIW	Field Blank	02/08/2012	02/10/2012

## **Batch Summary**

Analyte	Lab Matrix	Method	Prepared	Analyzed	Batch	Sequence
Hg	Water	EPA 1631	02/10/2012	02/13/2012	B120211	1200098



Analytical Lab Page 20 of 27 Client PM: Jay Perkins Client PO: 141391

## Sample Results

Sample	Analyte	Report Matrix	Fraction	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
<b>BioReactor 1 In</b> 1206023-01	<b>f</b> Hg	Influent	Т	229		15.3	40.8	ng/L	B120211	1200098
<b>BioReactor 2 E</b> 1206023-05	<b>ff</b> Hg	Effluent	Т	12.5		1.52	4.04	ng/L	B120211	1200098
<b>BioReactor 2 In</b> 1206023-03	<b>f</b> Hg	Influent	Т	67.6		3.03	8.08	ng/L	B120211	1200098
Hg Blk BioRead 1206023-02	e <b>tor 1 Inf</b> Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B120211	1200098
<b>Hg Blk BioRead</b> 1206023-06	e <b>tor 2 Eff</b> Hg	DIW	Т	0.15	U	0.15	0.41	ng/L	B120211	1200098
<b>Hg Blk BioRead</b> 1206023-04	e <b>tor 2 Inf</b> Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B120211	1200098



Analytical Lab Page 21 of 27 Client PM: Jay Perkins Client PO: 141391

## Accuracy & Precision Summary

Batch: B120211 Lab Matrix: Water Method: EPA 1631

Sample B120211-SRM1	Analyte Certified Reference Materia	Native al (1205004	Spike 1, NIST 1641d	Result	Units ion)	REC 8	Limits	RPD & Limits
	Hg	`	15.68	13.35	ng/L	85%	85-115	
B120211-MS1	<b>Matrix Spike (1206023-03)</b> Hg	67.57	303.0	366.7	ng/L	99%	71-125	
B120211-MSD1	Matrix Spike Duplicate (120	0 <b>6023-03)</b> 67.57	303.0	370.4	ng/L	100%	71-125	1% 24

## Method Blanks & Reporting Limits

Batch: B120211 Matrix: Water Method: EPA 1631 Analyte: Hg

Sample	Result	Units
B120211-BLK1	0.03	ng/L
B120211-BLK2	0.07	ng/L
B120211-BLK3	0.05	ng/L
B120211-BLK4	0.05	ng/L

 Average: 0.05
 Standard Deviation: 0.02
 MDL: 0.15

 Limit: 0.50
 Limit: 0.10
 MRL: 0.40

Project ID: DUK-HV1201 PM: Tiffany Stilwater



Analytical Lab Page 22 of 27 Client PM: Jay Perkins **Client PO: 141391** 

## **Instrument Calibration**

**Sequence:** 1200098 **Total Mercury and Mercury Speciation by CVAF** Instrument: THG-05

Method: EPA 163

Date: 02/13/2012 Analyte: Hg

Lab ID	True Value	Result	Units	REC	& Limits
1200098-IBL1		7.33	pg of Hg		
1200098-IBL2		6.20	pg of Hg		
1200098-IBL3		7.11	pg of Hg		
1200098-IBL4		6.03	pg of Hg		
1200098-CAL1	25.00	26.31	pg of Hg	105%	
1200098-CAL2	100.0	98.31	pg of Hg	98%	
1200098-CAL3	500.0	495.9	pg of Hg	99%	
1200098-CAL4	2500	2485	pg of Hg	99%	
1200098-CAL5	10000	9819	pg of Hg	98%	
1200098-ICV1	1568	1335	pg of Hg	85%	85-115
1200098-CCB1		8.51	pg of Hg		
1200098-CCV1	500.0	465.7	pg of Hg	93%	77-123
1200098-CCV2	500.0	459.3	pg of Hg	92%	77-123

**Project ID:** DUK-HV1201 **PM:** Tiffany Stilwater



Analytical Lab
Page 23 of 27

Client PM: Jay Perkins Client PO: 141391

## **Sample Containers**

Sampl	0: 1206023-01 le: BioReactor 1 Inf		Sample	Matrix: Influent Type: Sample		Receiv	ved: 02/10/2012				
	Container Bottle FLPE Hg-T	Size 500 mL	<b>Lot</b> 71511970 10	Preservation none	P-Lot n/a	Collected: 02/08/2012 Received: 02/10/2012 pH Ship. Cont. Cardboard Box  Collected: 02/08/2012 Received: 02/10/2012 pH Ship. Cont. Cardboard Box  Collected: 02/08/2012 Received: 02/10/2012 pH Ship. Cont. Cardboard Box  Collected: 02/08/2012 Received: 02/10/2012 pH Ship. Cont. Cardboard Box  Collected: 02/08/2012 Received: 02/10/2012 pH Ship. Cont. Cardboard Box  Collected: 02/08/2012 Received: 02/10/2012 pH Ship. Cont. Cardboard Box  Collected: 02/08/2012 Received: 02/10/2012 pH Ship. Cont. Cardboard Box					
	0: 1206023-02 le: Hg Blk BioReactor 1 Inf		Report Sample								
	Container Bottle FLPE Hg-T	Size 250 mL	<b>Lot</b> 71470160 10	Preservation none	P-Lot n/a	рН	Cardboard				
	): 1206023-03 le: BioReactor 2 Inf		Report Sample								
	Container Bottle FLPE Hg-T	Size 250 mL	Lot 71470160 10	Preservation none	P-Lot n/a	рН	Cardboard				
	): 1206023-04 le: Hg Blk BioReactor 2 Inf		•	Matrix: DIW  Type: Field Blank							
	Container Bottle FLPE Hg-T	Size 250 mL	<b>Lot</b> 71470160 10	Preservation none	P-Lot n/a	рН	Cardboard				
Lab ID: 1206023-05 Sample: BioReactor 2 Eff			Report Sample								
	Container Bottle FLPE Hg-T	Size 250 mL	<b>Lot</b> 71470160 10	Preservation none	P-Lot n/a	рН	Cardboard				
Lab ID: 1206023-06 Sample: Hg Blk BioReactor 2 Eff			-	Matrix: DIW  Type: Field Blank							
	Container Bottle FLPE Hg-T	Size 250 mL	Lot 71470160 10	Preservation none	P-Lot n/a	рН					

**Project ID:** DUK-HV1201 **PM:** Tiffany Stilwater



Analytical Lab Page 24 of 27 Client PM: Jay Perkins Client PO: 141391

## **Shipping Containers**

**Cardboard Box** 

**Received:** February 10, 2012 8:45 **Tracking No:** 4726 7966 8183 via FedEx

**Coolant Type:** None **Temperature:** ambient

Description: Cardboard Box Damaged in transit? No Returned to client? No Custody seals present? No Custody seals intact? No COC present? Yes

Analytical Lab CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM **Duke Energy Analytical Laboratory** Duke Energy<sub>s</sub>, Analytical Laboratory Use Only Mail Code MGO3A2 (Building 7405) ORDER# <sup>19</sup>Page 2 of 2 Sample Class OTHER Samples NC\_\_\_ 13339 Hagers Ferry Rd Originating 12020079 DISTRIBUTION Huntersville, N. C. 28078 ORIGINAL to LAB (704) 875-5245 0657 SAMPLE PROGRAM Ground COPY to CLIENT Fax: (704) 875-4349 **NPDES** Drinking Water Belews - FGD UST\_\_\_\_ Wis 13 (2011, b) We only Summer Brooks Rand RCRA Waste Cooler Temp (C) 2) Client: 4)Fax No: PO#141391 Bill Kennedy, Melonie Martin. Preserv.:1=HCL 2=H2SO4 3=HNO3 Wayne Chapman, Tom Johnson \* 4=ice 5=None 5 5)Business Unit: 6)Process: MR# <sup>16</sup>Analyses Required Mail Code: 8)Oper. Unit: Hg 1631 (sample 2nd week only) 9)Res. Type: 10)Reso. Center: Customer to complete all appropriate non-shaded areas. LAB USE ONLY Se Speciation Bottle 17Comp. 18Grab ID <sup>13</sup>Sample Description or ID 11Lab ID Date Time Signature \* BioReactor 1 Inf 28-12 1 Hg Blk BioReactor 1 Inf 1725 BioReactor 2 Inf 1 Hg Blk BioReactor 2 Inf 1 1320 BioReactor 2 Eff 1 703 Hg Blk BioReactor 2 Eff 1 Use the Bioreactor 2 Inf or EFF sample as the MS/MSD 1) Relinguished By Date/Time <sup>22</sup>Requested Turnaround 3) Relinguished By 4) Accepted By 5)Relinquished By Date/Time 6)Accepted By: 7)Relinquished By 8)Accepted By Date/Time 9)Seal/Locked By 10) Seal/Lock Opened By Date/Time \*Other \_\_\_\_\_ \* Add. Cost Will Apply 11)Seal/Locked B Date/Time 12)Seal/Lock Opened By Date/Time 2-16-12 Comments \* Metals=As, Ag, B, Cu, Cr, Ni, Se, Zn \*thomas.d.johnson@siemens.com

## CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

Duke Energy sm 1)Project Name Bele WWTS ( Bi-Moi		Fax: (704) 875-4349  ews - FGD 2)Phone No:			Analytical Laboratory Use Order # Matrix: OTHER  Logged By Date & Time 2 9 12 0657  AS&C Scoto Tem (6)								Samples Originating NC SC From SC  SAMPLE PROGRAM Ground Water NPDES Drinking Water UST								
2) Client: Bill	Kennedy,	Melonie Martin, Tom Johnson **	4)Fax No:		#133241		Con 15 Pres 2=H <sub>2</sub> S	oler T erv.:1 O <sub>4</sub> 3=	HNO	3				A Wa							
5)Business Unit: 8)Oper. Unit:		Process: Res. Type:	Mail Code: 10)Reso. Center:	MR#	stomer	to con	nplete all	5=1	Required	N.	1 3,4		4	3,4			vendor to	place filled baggies)			
LAB USE ONLY Se Spec							aded areas.	17 Comp. 16			- 245.1	Br (IC)	Metals*	Se, soluble			speciation -	AS&C (important to place filled bottle back into both baggies)			
WWTS (Bi-life property of the			escription or ID	Date	Time	0	Signature		β. Ω	TDS	Hg	B	Me	Mn,			Se,	Se AS8			
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	o sign & date	below - fill out from left to rig Date/Time	- 8 -/2	2) Accepted By	3 /2	Non	28	212	Date/T	ime	130	20			<del>-</del> 2:	<sup>2</sup> Reg	ueste	d Turi	naround		
3) Relinquished By 5)Relinquished By	Max 28-12 Date/Time 1530				4) Accepted By  April 2-8-12 153 6) Accepted By:  Date/Time								0	TANTI	aron	14 Da	4 Days				
7)Relinquished By  9)Seal/Locked By		2-9-/2 Date/Time		8)Accepted By: 10) Seal/Lock O	pened By				Date/Ti					r. IMPOF	e indicate desired turn	• 48 1	Hr				
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Comments * **********************************	rpw//cp-	R Mn TRM/IMS	=Ao Ao Cr Cu N	7										0	lease			6	12		

## CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

Duke Energy Analytical Laboratory					Analytical Laboratory Use Only												
En	ike ergy <sub>s</sub> ,	Mail Code MGO3A 13339 Hage Huntersville, (704) 8	Logged By	0200	79 Sample C	The Real Property lies and the Personal Property lies and the	NC SC	19Page 2 of 2 DISTRIBUTION ORIGINAL to LAB,									
(704) 875-5245 Fax: (704) 875-4349  1)Project Name Belews - FGD WWTS (2011, Bi-Weekly Sampling)  2)Phone No:				Ve CP		Date & Time 2-12	'	NPDES Drinking Water UST	COPY to	CLIENT							
2) Client:	Bill Kennedy	, Melonie Martin, an, Tom Johnson *	4)Fax No:	-	oks Ran #141391	1 1ºP	Cooler Te reserv.:1= I <sub>2</sub> SO <sub>4</sub> 3=	HNO <sub>3</sub>	H	RCRA Wa				T			
5)Business Unit:		i)Process:	Mail Code:	MR#		4=	Ice 5=N				5			1			
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LAB USE ONLY	Se Speciation Bott	le		Sampling	conducted: 2r	nd Wednesday each mor					31 Ne 2nd						
<sup>11</sup> Lab ID	ID	<sup>13</sup> Sample D	escription or ID	Date	Time	Signature	17 Comp.	18 Grab			Hg 1631 (sample		11	Т			
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mer to α						Use	the Bic	reacto	r 2 Inf	or EFF s	sample as the	MS/MS	D	1			
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) Relinquished By	Customer to sign & dat	e below - fill out from left to r												1			
Relinquished By	Man	2-8-22 Date/Time	1530	2) Agcepted By 4) Accepted By	ly 7	mor .		Date/Time		30	puno <sup>22</sup> Re	quested 1	Turnaroui	nd			
5)Relinquished By Date/Time		6)Accepted By: Date/Time							ti ti	4 Days 7 Days 48 Hr							
9)Seal/Locked By CPS 2-9-Date/Time 10)				8)Accepted By: Date/Time									Please indicate desired				
			10) Seal/Lock O	10) Seal/Lock Opened By Date/Time							Other* Add. Cost Will Apply						
11)Seal/Locked By					8)Accepted By:  Date/Time  10) Seal/Lock Opened By  Date/Time  12)Seal/Lock Opened By  Date/Time								2 -16-12				